Effect of Limestone Powder on Microstructure of Concrete

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Effect of Limestone Powder on Microstructure of Concrete

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Abstract: The effect of limestone powder on microstructure of concrete was studied by using mercury intrusion porosimetry (MIP), backscattering scanning electron(BSE), scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. The experimental results show that the compressive strength of concrete containing 100 kg/m^3 limestone powder can meet the strength requirement. Limestone powder has not pozzolanic activity; it is still unhydrated at the age of 28 days. But its filling effect can make the paste matrix and the interfacial transition zone between matrix and aggregate denser, which will improve the performance of concrete.

Key words: concrete; limestone powder; microstructure; filling effect; hydration characteristic

1 Introduction

High-performance concrete (HPC) mixtures contain a large volume of cement content and low *w/c* ratio; in addition, high workability can be achieved by using superplasticizer. In these mixtures, there is not available space to locate the hydration compounds and, as a result, a large volume of cement will remain unhydrated causing a nonrational use of resources and energy to produce concrete. In these concretes, the capillary pores become discontinuous relatively early and the improvement of the strength depends on the degree of hydration developed by the cementing materials. Moreover, the large volume of cement could induce an increase of cracking in the concrete structure. Replacing the expensive particles of cement by nonreactive economical particles of filler could offset these disadvantages[1-7].

The use of Portland cement containing limestone powder is a common practice in European countries, especially in France. Modern cements often incorporate several mineral admixtures, one of which is limestone powder. European standard EN 197 identifies two types of Portland limestone cements (PLC): Type Ⅱ/A-L containing 6%-20% and Type Ⅱ/B-L containing 21%-35%[8]. This type of cement is formulated to achieve certain goals in the technical, economic and ecological fields. Among the technical benefits are the increase of early strength, the control of bleeding in concrete with low cement content, and the low sensibility to the peak of curing[9]. The economic benefits are related to the possibility to obtain cement with a strength development similar to that of Portland cement at low production and investment costs per ton of cement[4]. The ecological advantages are the reduction of CO_2 and NO_x emissions per ton of cement manufactured and conservation of fossil fuels and mineral resources[10].

Although considerable researches have been carried out within the last 20 years on the use of limestone powder in ordinary concrete, they are still debating on what the effect of limestone powder on concrete and whether limestone powder will hydrate or not[11-16]. Some researches thought that the main effects of limestone powder are of physical nature. It causes a better packing of cement granular skeleton and a larger dispersion of cement grains.

Some researches thought that limestone powder acts as the crystallization nucleus for the precipitation of $Ca(OH)_2$. These simultaneous effects produce an acceleration of the hydration of cement grains. The objective of this study was to obtain the microstructure of the concrete containing limestone powder and to confirm its filling effect and hydration characteristics.

2 Experimental

The mixtures used were prepared with ordinary Portland cement PO 42.5 (Chinese standard GB 175-1999). Limestone powder, produced from carboniferous limestone of a very high purity (95% of $CaCO_3$ content), were added as filler. The particle size distributions of Portland

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cement and limestone powder measured by laser diffraction are shown in Fig.1. Obviously, the particle size of limestone powder is smaller than that of Portland cement. Fly ash, ground slag and superplasticizer Glenium Ace68 were also used. Coarse aggregate was crushed limestone with a maximum size of 20 mm.

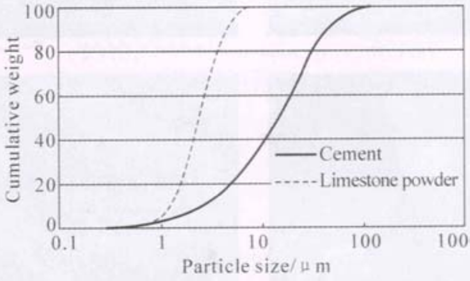


Fig.1 Particle size distributions of cement and limestone powder

The mix proportion design was based on the strength grade of concrete, C30, C40, C50 and C60. The mix proportions and properties of these concrete are shown in Table 1.

As illustrated in Table 1, although the four mixtures contain only 123-140 kg · m^{-3} water, their slumps are around 220 mm, which demonstrates that limestone powder can decrease the water demand. The compressive strength at the age of 28 d meet the strength requirement of concrete, which shows that limestone powder has not adverse effect on the strength of concrete.

Table 1 Mix proportions and properties of concrete

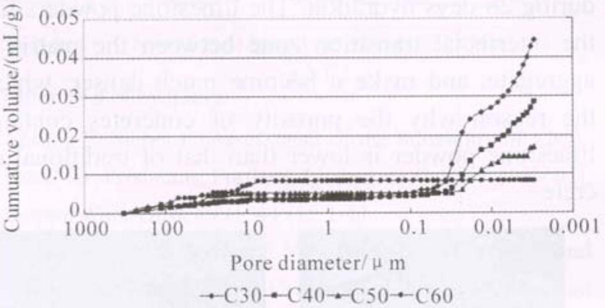
Mixture		C30	C40	C50	C60
Water/(kg · m^{-3})		123	123	133	140
Cement/(kg · m^{-3})		110	130	160	200
Limestone powder/(kg · m^{-3})		100	100	100	100
Fly ash/(kg · m^{-3})		60	60	80	0
Ground slag/(kg · m^{-3})		110	130	160	200
Fine aggregate/(kg · m^{-3})		948	912	820	831
Coarse aggregate/(kg · m^{-3})		989	988	1002	1017
Glenium Ace68/%		0.6	0.6	0.6	0.6
Slump/mm		225	220	220	225
Compressive strength/MPa	7 d	50.4	55.2	63.7	69.2
	28 d	61.8	66.5	74.2	81.1

3 Results and Discussion

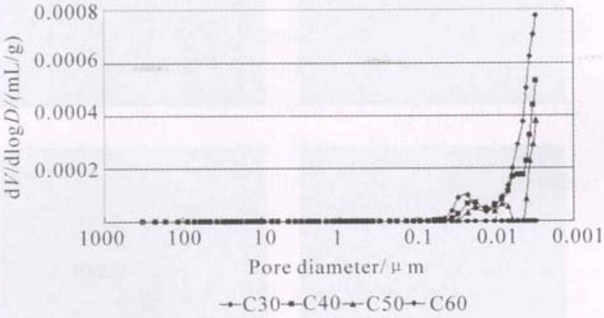
3.1 Mercury intrusion porosimetry (MIP) measurements

MIP was used to determine the pore size distribution of concrete. Porosity, pore size and pore shape are significantly influenced by mix proportion. Fig.2 shows the comparison of the four samples at 28 d, the results are plotted.

As expected, the total pore volume in the four concretes is much lower than that in traditional concrete. And with the increase of the strength grade, the total porosity is decreasing, especially for C60 sample. The critical pore diameters, defined as the peaks in the curves, giving the rate of mercury intrusion per change in pressure (differential curves), do not show a significant change. The size of most pores in the four concretes is lower than 50 nm, which does little or no harm to performance of concrete.



(a) Cumulative curves



(b) Differential curves

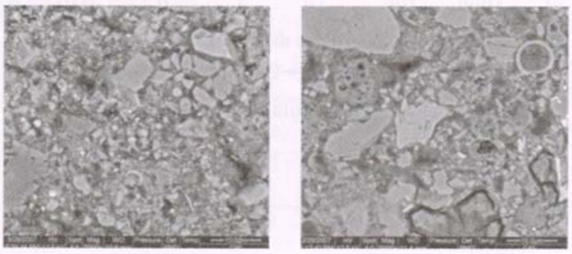
Fig.2 Pore size distribution of the four concretes at 28 d

3.2 Backscattering scanning electron (BSE) image acquisition and analysis

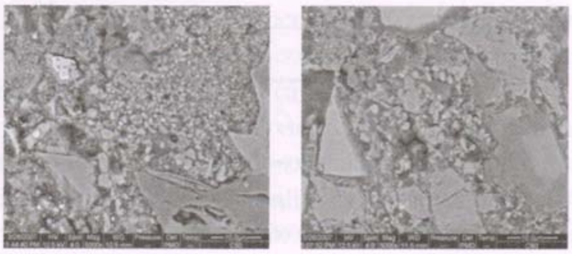
Scanning electron microscopy was used to determine the microstructure and the phase distribution of the samples. In order to obtain high quality images using a backscattering scanning electron detector, the samples had to be prepared carefully, including epoxy impregnation, cutting, grinding and polishing. The images were obtained in water vapour mode. In order to get a high contrast images for image analysis, an acceleration voltage around 12.5 kV was used. The magnification factor of region in each image was 5000. With the help of image analysis techniques, the grey-scale histogram of original image was used to distinguish the different phases. Within the grey-scale from 0 (black) to 256 (white), the pores, C-S-H gel, CH, limestone powder and anhydrous cement are identified respectively.

In order to make a quantitative comparison of the pore size distributions of the different samples, a 2D backscatter image analysis technique was developed and used. One of the BSE images from each mixture at the ages of 28 d is shown in Fig.3. Pore size and pore shape are significantly influenced by mix design. Little pores can be found in the four samples, especially in samples C60. There are some pores in samples C30, C40 and C50, which are mainly situated around aggregates. This is consistent with MIP measurements.

It can also be seen that small limestone particles exist in the pastes even after 28 days of hydration, the total amount of limestone powder is almost not changing during 28 days hydration. The limestone powders fill in the interfacial transition zone between the matrix and aggregate, and make it become much denser, which is the reason why the porosity of concretes containing limestone powder is lower than that of traditional concrete.



(a) C30 (b) C40



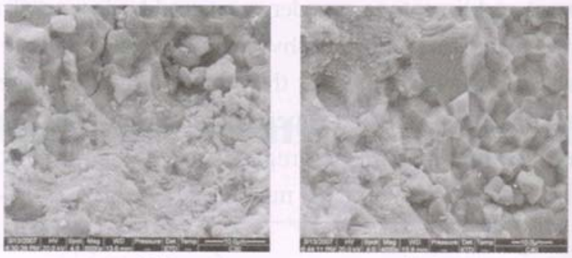
(c) C50 (d) C60

Fig.3 BSE images of the four samples at the age of 28 d

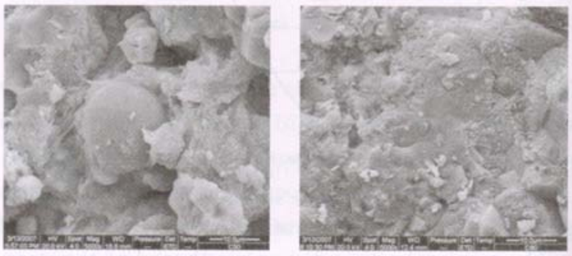
3.3 Scanning electron microscopy (SEM) analysis

Fragments of specimens broken off and washed with acetone were examined by SEM equipped with energy dispersed X-ray spectroscopy (EDS). This analysis was specially carried out to identify weather limestone powder can hydrate with hydration products of cement. SEM images from each mixture at the ages of 28 d is shown in Fig.4.

We can find the limestone powders presented in Fig.4. Comparing with the eroded spherical fly ash particle of sample C50 in Fig.4(c), the surface of limestone powders is still smooth, and there is no hydrate produced on their surface, which illustrates that limestone powder is unhydrated before 28 days. As shown in Fig.4, there are C-S-H gel, ettringite and $Ca(OH)_2$ crystals in samples C30, C40 and C50, but the microstructure of sample C60 is so dense that you can not distinguish what the hydration products they are, and which can prove its low porosity (MIP measurements) and the filling effect (BSE analysis) of limestone powder again.



(a) C30 (b) C40



(c) C50 (d) C60

Fig.4 SEM images at the age of 28 d

3.4 X-ray diffraction (XRD) analysis

XRD measurements were performed on a Philips X'Pert diffractometer equipped with a graphite monochromator using Cu Kα radiation and operating at 40 kV and 20 mA. Step scanning was used with a scan speed of 2° /min and sampling interval of 0.02° (2θ). XRD was used to identify the hydrates in the cement paste containing limestone powder. The mixture proportions of the three samples are shown in Table 2.

Table 2 Mixture proportions of the samples used in XRD analysis

Sample	$1^{\#}$	$2^{\#}$	$3^{\#}$
Water/g	203	117	117
Cement/g	700	256	198
Limestone powder/g	0	188	187
Fly ash/g	0	0	117
Ground slag/g	0	256	198
Glenium Ace68/g	3.6	4.2	4.2

Fig.5 shows the results of XRD analysis of hydrated cement pastes containing different proportions of limestone after 28 days of hydration. Fig.5a shows the hydrates of pure cement paste, which indicates that $Ca(OH)_2$ is the main hydration product. There is a $CaCO_3$ peak, which may be caused by carbonization. Fig.5b and Fig.5c present the hydrates of cement paste containing limestone powder. In the tow diagrams, ettringite, $Ca(OH)_2$ and mullite were detected by XRD, and the peak characterizing limestone, $CaCO_3$, appear also in the diffraction patterns, and it is the strongest peak. But we do not find any kinds of calcium monocarboaluminate hydrates such as $CaAl_2(CO_3)_2(OH)_4 \cdot 3H_2O$ (6.25Å, 6.50Å, 3.23Å, 7.21Å), $3CaO \cdot Al_2O_3 \cdot 3CaCO_3 \cdot 32H_2O$ (9.41Å, 2.51Å, 3.80Å) and $3CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11H_2O$ (7.57Å, 3.75Å, 2.85Å), which proves limestone powder is still unhydrated at the age of 28 d, as demonstrated in SEM analysis.

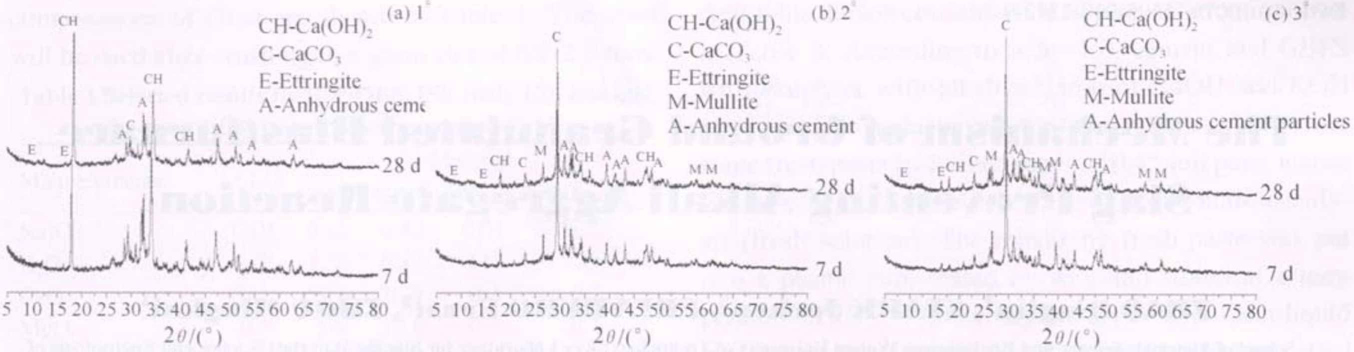


Fig.5 XRD analysis of hydrated cement pastes after 28 d of hydration

4 Conclusion

The compressive strength of concrete containing 100 kg/m^3 limestone powder can meet the strength requirement. The filling effect of limestone powder can make the paste matrix and the interfacial transition zone between matrix and aggregate denser, which can improve the performance of concrete. Limestone powder does not have pozzolanic properties, but it is still unhydrated at the age of 28 d.

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